Improvements in or relating to method of and apparatus for densifying dry powdered solids

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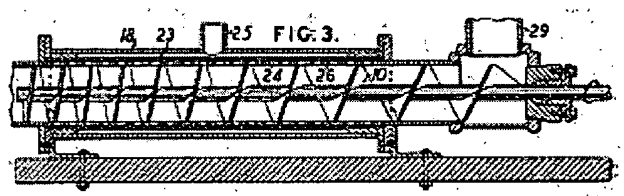
Abstract of **GB668394**

668,394. Screw pumps. MONSANTO CHEMICALS CO. April 19, 1949 [May 1, 1948], No 10331/49. Class 110(i) [Also in Group XVII] A device for densifying dry, powdered material, which contains occluded and included air, com- prises a casing containing a rotat- able screw conveyer 10 whose pitch increases progressively to- wards the exit end of the casing. The screw is surrounded by a freely-mounted, perforated, cylindrical member or wire screen 26 supporting a cylindrical canvas filter 24 which in turn is surrounded by a perforated, cylindrical member of wire screen 23 welded to the casing, the perforations in the member 26 being large compared with those in the member 23. The latter member is spaced from the wall 18 of the casing which is formed with a central pipe 25 connected to a source of vacuum. The member 26 may be omitted. The material is fed to the screw conveyer through an inlet 29 and is discharged from the exit end of the casing into packaging containers. Specification 649,896, [Group III], is referred to.



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Improvements in or relating to method of and apparatus for densifying dry powdered solids

Description of **GB668394**

COMPLETE SPECIFICATION
Improvements in or relating to Method of and Apparatus for
Densifying Dry Powdered Solids
We, MONSANTO CHEMICAL COMPANY, a corporation organised under the laws of the State of Delaware, United States of America, of 1700, South Second Street, City of St.

Louis, State of Missouri, United States of America (Assignees of WALTER M.DAVIS), do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :
The present invention relates to a method and apparatus for materially increasing the density of powdered substantially dry solids which contain relatively large quantities ofocc.luded or included air and/or other gases. The invention more particularly relates to a method and apparatus for materially increasing the density of finely divided or powdered silica aerogels and other similar aerogels which contain relatively large quantities of occluded or included air and/or other gases as a result of an antecedent pulverizing or grinding operation.

Silica aerogels, for example, those having extremely fine particles, often of microscopic size, are employed as flatting agents, pigments and the like in lacquer, paint and other coating compositions. In order to reduce the aerogel particles to the proper size consistent with such uses, it is necessary to subject the silica aerogel to a comminuting, grinding or pulverizing operation. However, when silica aerogel is pulverized or ground to the necessary degree of fineness, relatively large quantities of air and/or other gases become occluded or included therein and the resulting product has a very low density and is almost impossible to densify by vibrating means. Moreover, it is extremely difficult to densify or compress this material economically and efficiently.

On the other hand it is desirable to compress the material without causing the individual particles to agglomerate. Agglomeration of the particles is considered to be undesirable since the material loses some of its hiding power and fatting efficiency. If, during densification, the particles are caused to agglomerate they can be redispersed, but usually only by a prolonged and uneconomical grinding operation.

In the manufacture of carbon black, as, for example, lampblack, it is not generally necessary to pulverize or comminute the product since it is generally of the proper particle size as produced. However, relatively large quantities of air and/or other gases are generally occluded or included in the material as a result of the manufacturing operation. Such products and other powdered solids of substantially similar characteristics generally have a low density or low weight per unit of volume, which in some instances may be as low as 1.0 pound per cubic foot, the particular density in any case depending upon the manner in which the material has been manufactured, pulverized or ground and the nature of the material itself.

Because of the low density of such materials it is necessary to compress or densify them to as high a degree as is possible and practical prior to packaging and shipping otherwise the cost of packaging and shipping such materials would be prohibitive. However, the densification of such materials should be preferably accomplished without causing substantial agglomeration of the individual particles and in such a manner that the densified material is readily disintegrated into individual particles having substantially the same size as the original particles prior to densification. The present invention affords a simple, efficient and practical method and apparatus for producing such densified materials.

It is, accordingly, one object of the invention to provide a simple and efficient method and apparatus for increasing the density of substantially dry powdered solid materials which contain relatively large quantitiesof- occluded and included airand/or- other gaseswhereby a product isobtained which is economical to ship and package and which is easily disintegrated into its individual particles.

A further object of the invention is to compress anddensify bya continuous method finely divided or

pulverized silica aerogels and other pulverized aerogelscon- taining relatively large quantities of occluded air and/or other gases withoutsulestantially agglomerating the individual particles thereof.

A further object of the invention is to provide an apparatus forincreasing the density of powdered silica aerogels and other powdered aerogels and preparing aproduct having thecha,racteristics described in the foregoingobjects.

According to the present invention there.

isprovided amethod of increasing; the density ofsubstantiaji:y dry, powdered solids which contain occluded and included air, which comprises subjectingthe-powdered solids toe a compressiveforce while confining the powderedsolids to- a substantially fixed: volumespace, concurrentlyexhausting a-ir from.- the space without loss of the solids and then discharging thedensified solids
from the space.

The. present invention alsoprovides- a
compressingdevice for densifying substantially dry, powdered solids comprisinga cylindrical casing: a horizontally positioned rotatable screw conveyor extendinglongi- tudinally through;the- casing:. a freelymounted cylindrical shaped. filtering medium surrounding theconveyer - and insub- stantiallydose fitting relationshipt-herewith, a perforatedcylindrical: support <RTI ercompassing- the filteringmedium and fastened to the' casing in spacedrelationship. with' the inner; wails of thecasing means for rotating the conveyor, means for supplying dry powdered solids to one. end: of: the. conveyor, means. for exhaustingair from the casing,.

and- an exit opening for the solidsae the other end; of the conveyor.

The compressive force to which thedt powdered- solids issubjected - is- preferably a progressively increasing. compressive force.

The-air or gas which isoccluded orincluded' inthe finely divided orpowdered solid,. for example, a pulverized; silica aerogel; is exhausted therefrom:while it is confined in the fixed volume: space. In treating the materials as. described above,a coripression device which isdescribed in. greater detail hereinafter is preferably. employed.

A variety ofpowdered and substantially dry solid materials can be densified according.

to the practice of the invention as described herein. Thus,the' larapblack whichis prepared by the controlled oxidation or combustion of oil can be densified immediately after it is collected by the method and apparatus herein described.

Other substantially dry powdered solid materials such as dry and pulverized H-acid,
DDT and the like, which materials contain occluded or included air and/or other gases as a result of their method of preparation or as a result of a subsequent pulverizing, grinding or comminuting operation, may also be-successfully densified: by the practice of the present invention. The present invention is particularly directed to the densification of pulverized or powdered, substantially dry silica aerogels. and commercial lampblacks which contain relatively large quantities of occluded or included airand/or other gases, and, for convenience; the invention. will be: described with referenceto' the. densification of suchmaterial:
Pulverized silica aerogels of the type hereinbefore described may be preparedm a variety of ways. Ingeneral, a silica aerogel having. relatively coarse particles is prepared.

initially and is then pulverized or comminutedin. a suitable- apparatus such as a hammermill, ball mill, air attritionmill or the like until particles of the desired particle sizeare obtained. Such silica aerogel may be prepared,. for example, by first reacting an aqueous alkaline silicate solution such as. aq-ueous- sodium. silicate and an inorganic acid such as sulphuric acid to form a silicagel,
This: gel is first washed substantially free of inorganic saltsox electrolytes. and is then heated to raise- the pressure above the critical pressure of the liquid- in the- gel; which liquid may be water or some water-miscible organic liquid such. as ethanol which has been employed to replace the water inthe gel. The foregoing operationi & carried out- in a fixed volume system such as an autoclave and theheating is continued until substantially all of the liquid is converted to the vapor phase. The vaporized liquid is then slowly released. from the fixed volume.system at such a rate that the gel structure is not materially altered,

that is, the gel occupies substantially the same volume as the gel originally placed in the system
The product so obtained is usually in lump form after removal from the autoclave and is. then ground; or pulverized to the requisite particle size as hereinbefore described.

A similar silica aerogel can be prepared byautoclaving a suitable- acid-reacting silica organo-aquasol instead of a silica gel of the type described above: The procedure forantoclaviug suchan organo-aquasol is essentially thesame- as for theautoelaving of the silica geL Such an acid-reacting silica organo-aquasol can be prepared by first reacting an alkaline silicate such as sodium silicate and an inorganic acid such as sulphuric acid and- then adding thereto a water-miscible organic solvent such as ethanol or isopropanol in sufficient quantity to precipitate a substantial proportion of theelectrolyte formed during such reaction.

The precipitated electrolyte is then removed as by filtration, centrifuging or the like.

Suchorgro-aquasol and the method ofpreparing same is described in greater detailia British patent specification No. 649,896.

Such silica aerogels are adapted to be ground into very fine powders having aralorit x of particles with a particle size of10, microns orless. In this state they are quite fluffy and contain relatively large quantities of occluded or included air. If they are compressed they tend to return to their original volume when the pressure is released. Such powdered silica aerogels have a density varying, in general, between about1.0 pound to- 4 pounds per cubic foot and may be densified by the method and apparatus described herein to any practical density as, for example, a density of 5 to 9 pounds per cubic foot.

In the manufacture of lampblack of the controlled oxidation or combustion of oil, the productas obtained generally has a density of from about 4 to 8 pounds per cubic foot depending upon the method of preparation and the oil used. By the practice of the invention the density of such product can be increased to about 8 to 30potiads per cubic foot without substantially causing the individual particles toaXomerate.

It is also possible by the practice of the present invention to continuously densify fluffy materials such as the above and to obtain a product which is substantially uniform in density irrespective of normal fluctuations in density of material to bedeiisied.

The accompanying drawings illustrate a preferred embodiment of the apparatus used in preparing such densified products, inwhich Figure 1 represents a top plan view showing one embodiment of a screw compressor constructed in accordance with the present invention,
Figure 2 is a side elevation thereof,
Figure 3 is an enlarged longitudinal sectional view of the screw compressor casing or assembly taken along line 3-3 of
Figure1 and,
Figure 4- is an enlarged cross sectionalview taken along line 4-4 of Figure 3.

lieferring to the drawings, wherein similar numerals referto. similar parts, part 5 is a base for the screw compressor, which base may be fixed or mounted on wheels or castors or the like and is thus adapted to be moved from one position to another.

Firmly secured to base 5 is a housing. 6 on which is mounted a roller bearing assembly 7 and a ball bearing assembly 8. Screw shaft 9 carrying the screw 10 is mounted to rotate within the bearings 7 and 8 and has a chain driven sprocket 11 keyed thereto.

Sprocket 11 is driven by chain 12 through motor driven sprocket (not shown) or any other suitable device. The screw10 which is securely fastened to shaft 9 may be a screw of non-uniform pitch, the pitch of the screw diminishing towards the discharge end of the device as shown in Figure 3 or may be a screw of uniform pitch, although the construction shown is- preferred.

Referring specifically to Figures 3 and 4, which show the screw compressor assembly and casing therefor, section A is the discharge section comprising a cylindrical steel shell 13 and an annular steel flange 14 welded thereto, section B is the vacuum section, shown between the dotted lines in Figure.3, and section C is the feed section, where the dry powdered material to be compressed is fed to the apparatus. The entire assembly is firmly secured to angle irons 15 by screws 16, and the angleirons- 15 are in turn securely bolted to base 5 by stove bolts 17.

The vacuum section B, as shown in
Figures 3 and 4, comprises an outer cylindrical steel shell 18 having internallymounted annular collars 19 and externally mounted annular flanges 20 and 21 all welded thereto to form a firm unitary structure. Annular flange 20 is bolted to flange 14 of discharge section A and flange 21 is bolted to flange 22 of feed section C making the entire casing firm and unitary. Mounted within cylindrical shell 18 and welded to annular collars19 is a perforated cylindrical steel pla-te 23 having a large number of relatively small perforations. If desired this may be replaced by a strong cylindrical wire screen wrapped with relatively close helical windings of heavy steel wire having a sufficient diameter to prevent the wire screen from contacting the shell 18 when dry powdered material is conveyed through the vacuum sectionB by screw 10.'within the perforated cylindrical steel plate23 is a cylindrically shaped canvas cloth 24 or other suitable filtering cloth or medium which is of suchConstrUCtion as to substantially prevent penetration of dry powdered material therethrough. Fabric 24 provides a lapped fit with perforated plate 23 when air is exhausted from the vacuum section B through- pipe 25 by means of a suitable exhaust or vacuum system (not shown). Freely mounted within the cylindrical canvas cloth24 and abutting on the inner ends of casing section A & Bis a cylindrical perforated thin steel sheet 26 having relatively-large perforations therein as compared to perforatedcylindrical plate 23. This cylindrical perforated sheet may be replaced by a relatively light steel wire screen or may be omitted altogether, since it is employed primarily to prevent excessive wear on canvas cloth 24 by the rotating action of screw 10 and the abrasive effect of the dry powdered material being compressed. The inner surface of the perforated cylinder 26 forms a substantially continuous surface with the inner surface of the discharge section A and feed section C.

The feed section C as shown in Figures 1, 2 and 3 comprises an internally threaded tee 27 in which is screwed externally threaded cylindrical steel shells 28 and 29 and a standard stuffing box assembly 30 having a metal bushing 31 which seals the feed end of the screw compressor assembly and substantially prevents outside air from leaking into the assembly along shaft 9 when air is exhausted from the assembly through pipe 25. Cylindrical steel shell 28 is welded or otherwise firmly connected to annular flange 22. In order to facilitate the loading of the screw compressor, the cylindrical shell 29 is connected through a flexible rubber hose 32 or the like to a similar steel shell 33 having an annulardried flange 34 welded thereto. Positioned above flange 34 is a hopper 35 equipped with an annular drilled flange 36 similar to flange 34, which flanges may be screwed together as shown in Figures 1 and 2 to give a relatively airtight fit.

As is shown in Figures 3 and 4, screw 10 rotates within the screw compressor casing and is so corstructed and arranged as to provide a close fit with the inner surfaces of the fabric 24 or the cylinder 26. The clearance actually used is preferably of the order of 0.01 to 0.2 inches and in general should be such as to substantially prevent the build-up of layers of the powdered material between the outer edges of the screw 10 and the inner walls of the fabric 24 or cylinder 26. At the same time the clearance should be such as to prevent excessive wear on the screw and the inner walls of the cylinder 26. When cylinder 26 is omitted from the structure, the screw 10 should be in close fitting relationship with canvas cloth 24. The vane 37 at the discharge end of the screw 10 rides on the inner wall of steel shell 13 which thus furnishes additional support for the screw shaft 9.

The operation of the screw compressor is as follows:
Dry powdered silica aerogel or other powdered dry material stored in a hopper 35 is fed under gravity into feed section C and this material is forwarded by the rotating screw 10 toward the discharge end of the apparatus. Air is exhausted by means of a suitable vacuum apparatus through pipe 25 and thus a relatively large portion of occluded or included air is removed from the material.

Such air passes through the perforated cylindrical steel sheet 26, the canvas fabric 24 and the perforated cylindrical steel plate 23 while the powdered dry material is precluded by virtue of canvas fabric 24 from being exhausted therewith. The compressor reaches its maximum efficiency when the powdered material reaches the discharge section A since a highly effective seal is formed by the powdered material itself and air is substantially prevented from entering into the vacuum section from the discharge end of the compressor. When the air seal is formed at the discharge end in this manner the continued exhaustion of air through pipe 25 creates a suction within the screw compressor casing shown in Fig. 3 and this suction continuously drags powdered material from the hopper 35 into the feed section C where it is continuously picked up and forwarded by screw 10. When a nonuniform pitch screw 10 having its pitch diminishing towards the discharge end of the device, as shown in Figure 3, is used in forwarding the dry powdered material, air is not only exhausted therefrom as described above, but the screw exerts a progressively increasing compressive force on the material and compresses same. The air which is forced out of the material in this manner is exhausted through pipe 25 as described above. When the compressed and

densified material reaches the discharge section A and emerges therefrom it is collected in suitable packaging containers (not shown) and is then ready for shipment.

The degree to which the dry powdered material is densified or compressed depends upon several factors such as the speed at which the material is forwarded through the compressor, the order of vacuum or suction applied to the compressor assembly, the nature and density of the material itself, whether or not a non-uniform pitch screw, having its pitch diminishing towards the discharge end of the device is used and the length of the vacuum section. In general, the faster the screw is rotated the less the powdered material is compressed or densified. Conversely, if the screw is rotated at very low speeds the material may be compressed excessively and may be compacted to such an extent that the discharged product cannot be easily disintegrated into individual particles corresponding to those originally fed into the compressor. The degree to which the system is evacuated or the degree of vacuum employed is not particularly critical. In ordinary operation of the apparatus a vacuum of the order of 5" to 10" of mercury (this corresponds to an absolute pressure of 25" to20" of mercury) has given satisfactory results in the densification of powdered or pulverized silica aerogels and commercially produced lampblack.

A further understanding of the invention ,will be obtained from the following exampleswhich are intended to be illustrative but not
limitative of the scope of the invention..

EXAMPLE I
Dry powdared silica aerogel having a
density of about 2 pounds per cubic foot
was fed through a device of the construction shown in the accompanying drawings. A
non-uniform pitch screw as shown in Fig. 3
was employed to convey the material and
was rotated at a speed of 60 R.P.M. Airwas exhausted through pipe 25 so as to establish an absolute pressure in the vacuum section of24" of mercury. The materialissuing from the apparatus at the rate of 175 pounds per hour had a density of 7 pounds per cubic foot and was readily :dispersible in a ball mill with organic solvents,
water and the like into substantially the
same discrete particles as it contained priorto the densification.

EXAMPLE II
IDry powdered silica aerogel having a
density of about 3 pounds was densified as
described in Example I except that the
non-uniform pitch screw was rotated at a speed of 100R.P.M. The material which
was packaged as it emerged from the : apparatus at the rate of 225 pounds per
hour had a density of 8 pounds per cubic foot and was readily dispersible into its-ongmal individual discrete particles.

EXAMPLE III
Dry pulverized silica aerogel having a
density of about 4 pounds per cubic foot
was fed through an apparatus constructed
in accordance with the accompanying
drawings. A non-uniform pitch screw as -shown in Figure 3 was employed to convey
the material and was rotated at a speed of 140 R.P.M. Air was exhausted throughpipe 25 by means of a vacuum pump-so asgto maintain an absolute pressure of 25" of
mercury within the vacuum section B of the
apparatus. The material issuing from the
apparatus at the rate of 300 pounds per hour
had a density of about 8.5 pounds per
cubic foot and was readily dispersed into
its original individual particles.

EXAMPLE -IV
A substantially dry Grade 10 lampblack
(a true Germantown black), prepared by
bunting oil in an open pan and having a

pump was employed to exhaust air through pipe 25 and to maintain an absolute pressure of 24" of mercury within vacuum section B of the apparatus. The product which emerged from the discharge section A of the apparatus at the rate of 500 pounds per hour had a density of about 25 pounds per cubic foot and was readily dispersible in a ball mill with liquids such as organic solvents, water and the like into itsoriginal-individual discrete particles.

EXAMPLE V

A substantially dry Grade 2 lampblack prepared by burning an intimately mixed composition of air and atomized oil particles and having a density of-about 5 pounds per cubic foot was densified as described in Example IV except that the screw of non uniform pitch was rotated at a speed of 105 R.P.M. The densified product which issued from the apparatus at a rate of 350 pounds per hour had a density of about 19 pounds per cubic foot and could be readily dispersed into its original particle size.

It can be seen from the foregoing description and the above examples that dry powdered materials can be densified to a varying degree which is dependent upon the density and nature of the material being compressed or densified, the rate of feed and discharge, the extent to which the system is exhausted and upon other factors.

The material may be increased in density from about 2 to 6 fold by the practice of the invention.

What we claim is

1. A method of increasing the density of substantially dry, powdered solids which contain occluded and included air, which comprises subjecting the powdered solids to a compressive force while confining the powdered solids to a substantially fixed volume space, concurrently exhausting air from the space without loss of the solids and then discharging the densified solids from the space.

2. A method according to claim-1, wherein -said solids are continuously fed to said fixed volume space and are subjectedto- a progressively increasing compressive force while passing through said space and thereafter are continuously discharged from said space.

3. A method of increasing the density of substantially dry, powdered solids which contain occluded and included air which comprises feeding the -powdered solids into an- air-pervious, fixed-volume cylindrical passageway which is non-pervious to the solids and is surrounded by an enclosed chamber, subjecting' the powdered solids while they are in said passageway to the action of a rotating continuous helical surface which is adapted to convey the solids through the passageway and concurrently exhausting air from the chamber surrounding the passageway, whereby the powdered solids in the passageway are compressed and densified.

4. A method according to any one of the preceding claims, in which the solids are discharged from the fixed volume space or passageway before the individual particles thereof substantially agglomerate.

5. A method according to any one of the preceding claims in which the solids are confined within the fixed volume space or passageway until the density of the solids has increased to from twice to six times its original density.

6. A method according to any one of the preceding claims, in which the dry powdered solid is an aerogel, preferably a silica aerogel.

7. A method according to claim 6, in which the aerogel is a silica aerogel having a density of from 1 to 4 pounds per cubic foot and a major portion of particles less than 10 microns in diameter.

8. A method according to any one of claims 1 to 5 wherein the dry powdered solid is carbon black, preferably lampblack prepared by the controlled oxidation or combustion of oil.

9. A method, according to claim 8 in which the carbon black has a density of from 4 to 8 pounds per cubic foot.

10. A compressing device for densifying substantially dry powdered solids comprising a cylindrical casing, a horizontally positioned rotatable screw conveyor extending longitudinally through the casing, a freely mounted cylindrical shaped filtering medium surrounding the conveyor and in substantially close fitting relationship therewith, a perforated cylindrical support encompassing the filtering medium and fastened to the casing in spaced relationship with the inner walls of the casing, means for rotating the conveyor, means -for supplying dry powdered solids to one end of the conveyor, means for exhausting air from the casing, and an exit opening for the solids at the other end of the conveyor.

11. A device according to claim 10 in which the filtering medium is a fabric which is pervious to air but impervious to said solids.

12. A device according to either of claims 10 or 11 in which a loosely mounted perforated cylindrical insert liner encompasses the conveyor and is located between-the conveyor and said filtering medium.

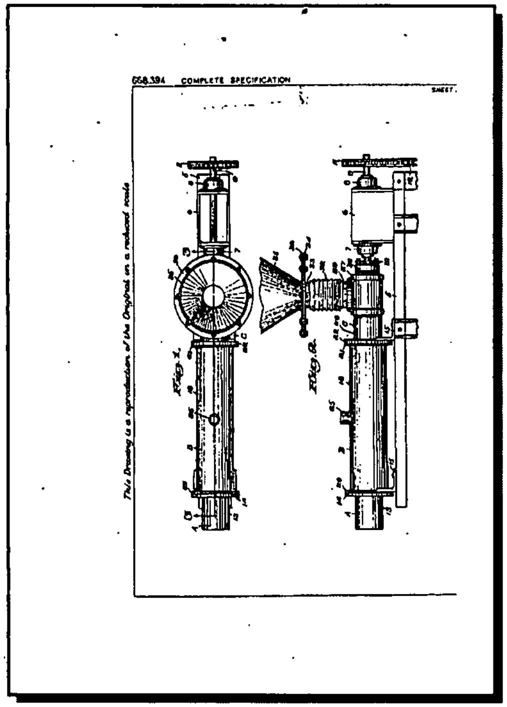
13. A device according to any one of claims 10 to 12 in which the screw conveyor is a screw of non-uniform pitch, the pitch of which diminishes progressively toward the discharge end of the casing.

14. A device 'according to any one of claims 10 to 12 in which the means for supplying dry powdered solids to one end of the conveyor includes an opening in the top portion of the feed end of the casing, the means for exhausting air from the casing includes an opening positioned in the central portion of the casing and the exit opening is an opening in the end of the casing of substantially the same diameter as the screw conveyor.

15. A compressing device for densifying substantially dry powdered solids comprising a horizontally positioned cylindrical casing having alongitudinal portion between the ends thereof of enlarged diameter, a rotatable screw of non-uniform pitch, the pitch diminishing towards the discharge end of the device, for conveying powdered solids along the longitudinal axis of the casing, an inlet opening to the screw in the casing, an outlet opening from the screw in the casing, an air outlet in the longitudinal portion of enlarged diameter in the casing through which air within the casing is exhausted, a perforated cylindrical insert liner encompassing the screw and loosely mounted within the longitudinal portion of enlarged diameter, a filtering fabric freely mounted within the longitudinal portion of enlarged diameter and encompassing the liner, which fabric prevents egress of solids through the air outlet, a perforated cylindrical support encompassing the filtering fabric and fastened to the casing, the casing being spaced from the support, means for rotating the screw and means for exhausting air from the casing.

16. A compressing device for densifying substantially dry, powdered solids constructed and adapted to operate substantially as herein described with reference to the embodiments shown in the accompanying drawings.

17. A method of increasing the density of substantially dry, powdered solids substantially as herein described with reference to any one of the examples.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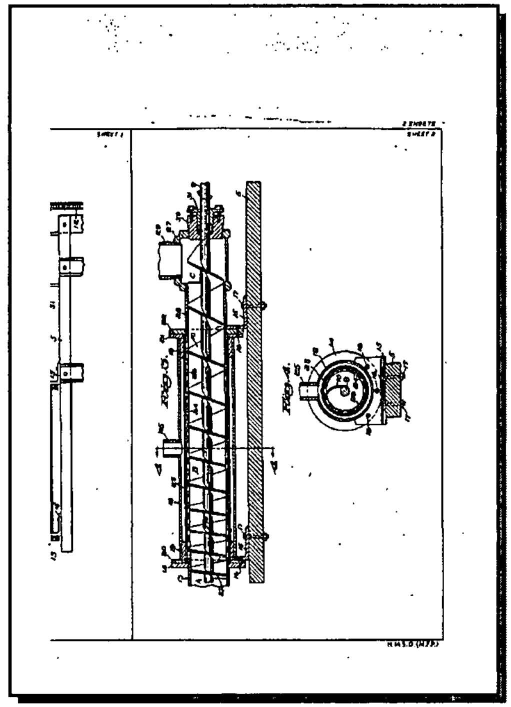
2 SHEETS
SHEET 2
SHEET 1